Terminal Residues of β-Cyfluthrin in Cotton

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Abstract

Degradation behavior of β–cyfluthrin in cotton seed, lint and soil under crop was studied, by conducting a field experiment at the Research Farm of CCS HAU, Hisar. The mean deposits were found to be 0.005 and 0.022 mg kg^{-1} in cotton lint at the time of harvest for minimum effective (18 g a.i ha^{-1}) and double effective dose (37.5 g a.i ha^{-1}), respectively. Average residues in cotton lint were found below MRL value at single dose and nearly equal to MRL value at double dose application. In cotton seed oil, the residues were observed to be below MRL at both the doses. In soil the *β* –cyfluthrin residues maintain little amount at harvest time.

Keywords

Residues; β –Cyfluthrin; Cotton Seed; Lint; Soil; MRL

Introduction

Cotton termed as "The King of Fibres and a crop of prosperity", having a great impact on men and matter, is an industrial commodity of worldwide importance. It is a variety of plants of the genus Gossypium, belonging to the Malvacae family. Out of about 50 species of cotton plants in the world, only four have been domestically cultivated for cotton fibres.

The areas under cotton production in the world are estimated at around 30-31 million hectares. India is the largest area under cotton production. China is the largest producer of cotton in the world whereas India stands at second position. Interestingly, although China with almost half the area under cotton production compared to India, but produces more than 2½ times yield (kg per hectare) of cotton as compared to India.

Cotton is one of the principal crops of India and plays a vital role in the country's economic growth by providing substantial employment and making significant contributions to export earnings. The cotton cultivation sector not only is engaged in around 6 million farmers, but also involved in another about 40 to 50 million people relating to cotton cultivation, cotton trade and its processing (Anonymous, 2012). Low productivity of cotton may be attributed to both biotic and abiotic stresses. Among the biotic stresses, insect pests are known to cause heavy loss to cotton resulting in drastic reduction in yield. Unfortunately, the pest spectrum of cotton is quite complex and is attacked by 1,326 species of insect pests from sowing to maturity (Santhum, 1997). Crop losses in cotton have been reported due to several pests.Among these nine are key pests in India out of which the tobacco caterpillar, *Spodoptera litura* (Fab.) is apolyphagous lepidopterous pest causing damage to 112 crop species throughout the country such as cotton, tobacco, chilli, groundnut, castor etc. (Lefroy, 1908). Other chief species include *Earias vittella* (30-40%), *Pectinophora gossypiella* (20-95%) (Panwar, 1995) and *Helicoverpa armigera* (20-80%) (Monga and Jeyakumar, 2002). Several potent pesticides have been recommended for managing these pests on this crop which consumes around 50% of pesticides in India Singh *et al.*(2004) and accounts for 40% of the total production cost and ranks first in terms of pesticide consumption, Dudani and Sengupta (1992).

Indiscriminate use of pesticides to combat insect pests led to resistance and also development of resurgence. To overcome these problems,several new insecticides with new chemistry have been tested in various parts of the world. The compound β-cyfluthrin, [cyano(4-fluoro-3-phenoxyphenyl) methy l3-(2,2- dichloroethenyl)-2,2-dimethylcyclopropanecarboxylate] belonging to synthetic pyrethroid group, acts as a contact and stomach poison. It gives wide spectrum activity against lepidoptera, coleoptera and hemiptera pests and various workers have studied residues of β-cyfluthrin in rice , Naini *et al*. (2003), cotton Battu *et al*. (1999), Mukherjee *et*

al.(2002), brinjal (Dikshit, 2001; Sinha and Gopal (2002) and Mandal *et al*. 2010), okra (Dikshit *et al*. 2002) and tomato (Dikshit *et al*. 2003 and Dharumarajan *et al*. 2009). As very scanty work has been carried out to know the behaviour of β -cyfluthrin on cotton under Indian conditions, Therefore the present study was carried out to evaluate the residues of β -cyfluthrin in cotton lint, seed oil and soil under crop at harvest.

Materials and Methods

All the solvents were of analytical grade and glass distilled before use. β –cyfluthrin was purchased from local market. Standard stock solution (1,000 µg/ml) was prepared in acetone. Lower dilutions were prepared by taking required aliquot from the stock solution and diluting it with n-hexane. An aqueous solution of NaCl (10%) was partitioned twice with dichloromethane to remove the impurities. Sodium sulfate was washed with acetone, dried at room temperature, and then activated at 110◦C for 4 h before use.

The cotton crop (variety H-1226) was raised at the research farm of CCS Haryana Agricultural University, Hisar during *kharif* season of year 2010 following recommended agronomic practices. The Plot size was taken as 25 m^2 each. There were three replications for each treatment (i.e control, recommended and double the recommended dosages) the recommended dosages arranged in a randomized block design (RBD). β – cyfluthrin was applied in the field at the time of flower initiation stage at two different doses. Before spraying, cotton plants in all plots/replicates were tagged and sprayed with recommended doses (T_1) consisting of 18.75 g $a.i.ha^{-1}$ and the doubled recommended dose (T_2) consisting of 37.50 g $a.i.ha^{-1}$ and a control where no pesticide was applied. Cotton lint, seeds and soil samples from top 15 cm of soil profile were collected at the time of harvest and then processed.

The cotton seed samples were air dried and delint to get cotton seed and lint to analyze them separately. Representative cotton seed sample (10 g) and cotton lint (5g), was extracted by Soxhlet apparatus using 200 ml acetonitrile for 8 h. The extract was then filtered, transferred in separatory funnel and diluted it with 10% sodium chloride solution. The extract was partitioned twice with hexane (100 and 100 ml) followed by partitioning twice with dichloromethane (100 and 100 ml) by vigrous shaking. The organic phases were combined and then concentrated to about 5 ml on a rotary vacuum evaporator at 50-55°C. For clean–up, glass column (60 cm x 22 mm i.d) was packed compactly with silica gel in between two layers of anhydrous sodium sulphate. The column was prewetted with hexane firstly and then the concentrated extract was loaded in the column. The column was eluted with 125 ml solution of dichloromethane: acetone (1:1 v/v). The cleaned extract was evaporated to dryness and finally dissolved in 2ml n-hexane for analysis by GC.

Soil samples were extracted as per method of Kumari et al.(2008). To the well-ground, sieved and representative soil sample (20 g), 0.5 ml ammonia solution was added and kept for half an hour. Ten grams of anhydrous sodium sulfate, 0.3 g Florisil and 0.3 g activated charcoal were added and mixed properly. The homogenized sample was packed compactly in a glass column (60 cm × 22 mm i.d.) in between two layers of anhydrous sodium sulfate. The column was eluted with 150 ml solution of hexane: acetone (1:1 *v/v)*. Eluate was concentrated to 5 ml on a rotary vacuum evaporator at 40◦C followed by gas manifold evaporator to dryness. Final volume of the concentrated extract was reconstituted by adding n-hexane up to 2 ml and analyzed by GC.

The cleaned extracts were analysed on GC Shimadzu 2010, Model, equipped with capillary column using a Ni^{63} electron capture detector (ECD). Operating conditions were: fused silica column: 30 m x 0.32 mm i.d, coated with 5% diphenyl/ 95% dimethyl silicone, 0.25 µm film thickness (supelco SPB-5), with split injection system.

N_2 was used as carrier gas at a linear gas velocity of 2 mL min−1 throughcolumn and made up gas 60 mL min^{-1}. The injection port maitained at 280°C and the oven temperature was 150°C for 5 min. then at 8°C min^{-1} to 190°C for 2 min, and finally at 15°C min^{-1} to 280°C for 10 min. The detector temperature, 300°C was used for estimation. Retention time was 20.72 min. Limit of detection and determination/quantitation were 0.002 and 0.005 mg kg−1, respectively.

The efficiency of the method was evaluated by carrying out recovery experiments. The percent recoveries of β -cyfluthrin in cotton lint were 81.60 and 83.77 and in

cotton seed oil were 81.05 and 82.31 at 0.25 and 0.50 mg kg^{-1} level respectively (TABLE 1).

TABLE 1 PERCENT RECOVERY OF B –CYFLUTHRIN IN COTTON LINT AND SEED OIL

Substrate	Level of Fortification ($mgkg^{-1}$)	% Recovery
Cotton Lint	0.25	81.60 ± 4.85
	0.50	83.77 ± 2.16
Cotton Seed Oil	0.25	81.05 ± 1.95
	0.50	82.31 ± 1.80

*Mean±SD of three replicates

Results and Discussion

TABLE 2 RESIDUES (mg kg^{-1}) OF B –CYFLUTHRIN IN SOIL, COTTON LINT AND COTTON SEED OIL AT SINGLE DOSE

Commodity	**Residues ($mgkg^{-1}$)**	
	Single Dose (18.75 g a.i ha^{-1})	Double Dose (37.50g a.i ha^{-1})
Cotton Lint	0.005 ± 0.004	0.022 ± 0.005
Cotton Seed Oil	BDL	0.010± 0.004
Soil (Harvest)	0.052 ± 0.013	0.127 ± 0.022

MRL: Cotton seed 0.020 mgkg-1, BDL: 0.005 mgkg-1

The overall results of the analysis of cotton lint and seed following the application of β -cyfluthrin @ 18.75 g a.i. ha^{-1} and 37.50 g a.i. ha^{-1} are presented in TABLE 2. The average deposits of β -cyfluthrin were found to be 0.005 and 0.022 mg kg^{-1} on cotton lint on harvest at minimum effective and the doubled effective dosages, respectively. In case of seed oil,the residues were found below detectable level for minimum effective dose while 0.010 mg kg^{-1} for the doubled effective dosages. In soil 0.052 and 0.127 mg kg^{-1} residues were found at single and double dose, respectively. The result revealed that the applied dose persisted in soil at the time of harvest. As the residues reached below MRL value hence safe for human consumption. The results were similar be similar to findings of Raj *et al*.1990, who reported that deltamethrin did not leave any residues in cotton seed, oil and lint. Fenvalrate and cypermethrin residues were below the maximum residue limit of 0.2 ppm in cotton seed. Residues were considerably lower in second picking for all the insecticides. Residues of synthetic pyrethroid were much lower than those of carbaryl and levels fell sharply during second picking of bolls. Residues levels were also found below MRL value for all the insecticide treatments and posed no toxicological hazard when presented in cotton seed oil by Gupta *et al*. (1990). Battu *et al*. (1999) estimated that residues of synthetic pyrethroids in cotton seed and lint. No residues were found in case of cotton seed but residues of cypermethrin and fenvalrate were detected in case of lint. The residues dissipated with the half-life of 2.4 and 2.6 days persisted for 5 days only. The mean initial deposits of β-cyfluthrin were 0.12 and 0.23 mg kg^{-1} on the okra fruits following 3rd application with respect to β-cyfluthrin at 18 and 36 g a.i. ha^{-1}. These deposits were disspated to 0.02 and 0.06 mg kg^{-1} after 1 day at single and double dosages respectively, thereby showing a loss of about 83.33 and 73.91 per cent. These residues of β-cyfluthrin reached below its determination limit of 0.01 mg kg^{-1} in 3 and 5 days at single and double dosages, respectively (Sahoo et al.2012). Mature mango fruits at harvest were free from residues of β -cyfluthrin by Mohapatra *et al*. (2011). Similar results were shown by Singh and Singh (2007).

Mandal *et al*. (2010) reported that soil samples under brinjal crop did not show the presence of β -cyfluthrin at their detection limit of 0.01 mg kg^{-1} when collected at the time of harvest. Half-life ($T_{1/2}$) values of β -cyfluthrin were observed to be 1.74 and 1.39 days, respectively, when applied @ 18 and 36 g a.i. ha^{-1}. Vig *et al*. (2001) observed that cypermethrin was more persistent as compared to deltamethrin under cotton crop in soil and up to 63–67% of cypermethrin residues were detected in 1995 after 15 days and up to 54.9% in 1998 between 2–10 days after treatment. No residues were detected on 105 day. Similar results were reported by Dikshit et al.(2002).

Conclusion

The applied dose is effective for the pests as the residues are persisting in the soil. As the residues are below MRL value hence safe for the human consumption. Residues are presented in Cotton lint at higher dose only at higher dose, but its safety couldn't be assessed due to non availability of MRL value.

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